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Plumbrook Hypersonic Tunnel Facility Graphite Furnace Degradation Mechanisms

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March 1999

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Acknowledgments

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PLUMBROOK HYPERSONIC TUNNEL FACILITY GRAPHITE FURNACE DEGRADATION MECHANISMS

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SUMMARY

A recent rebuild revealed extensive degradation to the large graphite induction furnace in the Hypersonic Tunnel Facility (HTF). This damage to the graphite blocks and insulating felt is examined and modeled with a thermochemical equilibrium code. The primary reactions appear to be with water vapor and the nitrogen purge gas. Based on these conclusions, several changes are recommended. An inert purge gas (e.g., argon or helium) and controlling and monitoring water vapor to about 10 ppm should decrease the damage substantially.

INTRODUCTION

The NASA Plumbrook Hypersonic Tunnel Facility was reactivated in 1990 to 1994. The facility has been described in detail in a recent report (ref. 1). It is a unique propulsion test facility which can simulate up to Mach 7 flight conditions with true air composition.

A key component is the large graphite induction furnace, consisting of a vertical stack of fifteen large graphite susceptors. A schematic diagram of the furnace is shown in figure 1. The approximate size of the furnace is 9 ft diameter x 40 ft high. Each susceptor (UCAR PGX Molded Graphite) is ~6 ft diameter by 2 ft high with 1945 vertical holes, ranging from 0.75 to 1.125 in. diameter. The weights of each block range from 2900 to 4600 lbs. A photograph is shown in figure 2(a). Between the graphite blocks and the induction coils is graphite felt insulation and silicon carbide (SiC) tile. The graphite felt insulation is illustrated in figure 2(b). The SiC tiles have numerous holes for pressure equalization.

The furnace takes about one week to reach temperature in standby operation. In standby operation, a flow of about 50 cfm nitrogen (N_2), preheated to ~200 °F and at a pressure of 5 to 7 psig is passed through the system at 3000 to 4500 °F and out the stack. Nitrogen is supplied from an adjacent tank car. Water vapor (H_2O), carbon monoxide (CO), and oxygen (O_2) are monitored.

During a test this furnace is used to heat nitrogen to 3500 to 4500 °F at a pressure of 300 to 1200 psig and a flow rate of 50 to 200 lb/sec, which is then blended with oxygen to make synthetic air. This is expanded through a nozzle into the test section (ref. 1). Tests typically last 1 to 5 min.

In September 1996, the heater isolation valve failed during a test. Immediately after this incident the furnace was shut down and allowed to cool. However an inrush of air and water likely occurred.

After the incident the facility was disassembled and the damage assessed. Substantial degradation of the graphite susceptors and the insulating graphite felt was noted. Evidence suggests this degradation occurred primarily during the purging and only partially as a result of the incident. The purpose of this report is to explore the chemical reactions causing the degradation occurring during normal standby operation and suggest possible solutions.

ASSESSMENT OF FURNACE CONDITION

Table I lists the observed recession and condition of the blocks and table II lists the condition of the graphite felt (ref. 2). The location of each block can be seen in figure 1. We have used a graphite density of 109 lb/ft³.

An estimated 5 to 10 percent of the felt was consumed or about 218 lbs. From the graphite blocks alone, about 3083 lbs was lost. This combines with the amount of consumed felt for a total of 3301 lbs (1.5×10^6 gm). Most of the consumption appears to be adjacent to and immediately above the large recirculating tube coming off the midpoint of the furnace, as shown in figure 1. Vertical gradients would make these the hotter blocks, since they are near the

furnace center. Horizontal gradients through the blocks should be minimal and the block temperature on the outside edges should be close to that in the interior after the furnace reaches temperature. However, in the event of a leak from either the induction coils or furnace ports, these edges would see more impurity gases.

The estimated time at temperature between the two rebuilds is given in table III (ref. 2).

Clearly a large amount of graphite was consumed over a relatively short operation period. This has three serious effects:

1. Decrease in performance.
2. Release of toxic gases through the vent
3. Clogging of lines and 'fire-flies' in the synthetic air.

PROBABLE CHEMICAL REACTIONS

The first issue to address is the cause of this large amount of graphite consumption. One scenario is the possible back diffusion of air and water after the incident; a second is the graphite reacting with the purge gases.

Back-diffusion of air into the furnace after the incident is clearly a serious issue, since carbon reacts readily with oxygen at elevated temperatures. Also, molten metal deposited on the top block, which is indicative of significant flashback.

However it is unlikely that much back-diffusion and block consumption due to the incident occurred for several reasons:

1. The positive pressure of nitrogen flowing outward would limit the backflow of oxygen inward.
2. The inner core of the blocks was not attacked, which would have been 'seen' first by any backflow.
3. Attack was primarily in two modes: (I) Reduction in diameter of the upper graphite blocks (II) 'Furrowing' or deep groove formation and consumption of the felt. Figure 2(b) is a photo of one of these grooves. These would both be the last to 'see' an inrush of air.
4. During normal purge procedure, the following observations were made, which support graphite degradation during this procedure.
 - a. On-line gas analyzers indicated increases in $H_2O(g)$ tracked with those in $CO(g)$, as will be discussed below. This indicated that the $H_2O(g)$ in the furnace was reacting with the graphite to form $CO(g)$ during the purge.
 - b. Dusting was observed in the vent, also indicating degradation of the graphite blocks and felt during the purge.

Thus evidence indicates that reactions with the purge gas led to the majority of the observed graphite degradation. This will be the focus of this report.

REACTIONS OF GRAPHITE WITH $N_2(g)$

Commercial graphite furnaces are generally not purged with nitrogen above 2000 K (3141 °F) (ref. 3). In order to determine the possible reactions, we used a standard free energy minimization computer code (ref. 4). This code takes an assemblage of reactants and determines the product composition which gives a minimum free energy. At these high temperatures, it is reasonable to assume no kinetic barriers to thermodynamic equilibrium. Hence thermodynamics is an excellent tool for predicting reactions. For various other applications it has been reported that product vapor pressures of 10^{-6} bar or greater lead to component degradation (refs. 5 and 6).

To correctly apply this code to the problem at hand, the proper choice of reactant amounts is necessary. We fix the chemical activity of graphite equal to one (essentially an infinite piece of graphite) and put in fixed amounts of nitrogen. A total pressure of 1.4 bar (6 psig or 20.7 psia) was used and an input of 1.4 moles of nitrogen. This gives a constant pressure of 1.4 bar nitrogen above the graphite in the final products. However a portion of the nitrogen reacts to form such carbon-nitrogen species as $CN(g)$ and $(CN)_2(g)$ at reasonably large vapor pressures, as illustrated in figure 3. This suggests the key reactions are:





Note also the formation of such species as C(g), C₂(g), and C₃(g). This is from the direct vaporization of carbon, which will always occur at high temperatures, regardless of the atmosphere.

REACTIONS OF GRAPHITE WITH H₂O(g)/N₂(g)

Figure 4 illustrates the output of several of the gas analyzers at position N28 on the furnace (see fig. 1). These data are very significant. Figure 4(a) indicates a substantial amount of water in the gas stream (>100 ppm). Variations in this amount of water track very closely with the amount of CO(g) (fig. 4(b)). This immediately suggests the oxidation of carbon by water vapor:



This is the well-know “water-gas” reaction and goes to completion at high temperatures.

The measured oxygen content of the furnace was quite low (fig. 4(c)), as expected. It is also likely that there are variations in the amount of H₂O(g) throughout this furnace.

Examining the thermochemical equilibria between carbon and H₂O(g)/N₂(g) is more complex than that with N₂(g) alone. Carbon and water vapor cannot be in equilibrium at high temperatures, i.e., reaction (2) goes to completion. Any amount of water vapor in a system with carbon is completely consumed. Thus we model a total pressure of 1.4 bar with 1.4 bar nitrogen and initial water vapor levels of 1, 10, 100, and 1000 ppm. This is illustrated in figures 5(a) to (d), respectively. The key reaction is now:



Note that as water vapor content of the system increases, reaction (3) becomes more important. This can be seen in figures 5(a) to (d), where the amount of CO(g) and HCN(g) increase with water vapor content. Note also the presence of HCN(g) and HNC(g). HCN is hydrogen cyanide and HNC is an isomer—nitrogen hydride carbide (ref. 7).

SOURCES OF WATER IN THE SYSTEM

It is likely that the formation of the above species leads to the consumption of the 3301 lbs (1.5×10⁶ gm) of carbon. The first issue to be addressed is if we have sufficient water in the system to do this. Consider again reaction (3). To volatilize one mole of carbon, we need one third mole of water vapor. We have 1.25×10⁵ moles of carbon which requires 4.17×10⁴ moles of water vapor. Is it possible to have this much water vapor in this chamber? First consider the moisture meter, which indicates a constant 100 ppm of water in the purge gas. This means that of the gas that flows past the graphite, 100 ppm of it is water. If 50 ft³/min flow past the graphite for six months, this means that ~1.3×10⁷ ft³ will have flowed past the graphite. Of this, 1.3×10³ ft³ is water, which is ~2×10⁶ moles of water—more than enough for the predicted reactions.

At issue is exact source of the water vapor. The nitrogen from the tank car is reasonably dry. This leaves adsorbed water in the system or a leak in the water-cooled induction coils. In a vessel this large with so much internal surface area, adsorbed water is certainly an issue. The majority of the internal surface area is on the felt. Table III lists the properties of the felt.

To determine if a monolayer of water adsorbed on the felt can account for this amount of water, let us assume that 10¹⁵ molecules of water/cm² make a monolayer (ref. 9). In 2900 lbs of felt, this corresponds to only 22 moles of water. We need at least 4.17×10⁴ moles of water to account for the observed consumption of carbon. Other surfaces may have adsorbed water, but it is unlikely that they can contribute the additional needed amount. This suggests that the water comes from a leak—either from the exterior or from the water-cooled induction coils.

TRANSPORT CONTROLLED CONSUMPTION OF GRAPHITE

As indicated, most of the consumption is adjacent to and immediately above the recirculating pipe at the mid-point of the furnace (fig. 1). Vertically, this is the hottest part of the furnace. Horizontally, there is probably a fairly constant temperature through the blocks. However, possible water leaks from either the induction coils and/or furnace ports would be closest to the outer edges of the graphite.

We know that as soon as a water molecule strikes a graphite surface, it will react. The problem then becomes one of gas phase transport. Total graphite consumption rates are dependent on how rapidly the product gases can be transported away from the graphite surfaces. This may explain why the most consumption is in the vicinity of the recirculating pipe which draws product gases away from the graphite.

The gas transport paths are complex and a number of processes are occurring. For the graphite blocks:

1. CO(g) and C-N gaseous species are formed at the block surfaces
2. These species diffuse radially outward through a boundary layer.
3. These species diffuse radially outward through the graphite felt.
4. These species diffuse radially outward through the silicon carbide

For the graphite felt:

1. CO(g) and C-N gaseous species are formed on the fiber surfaces.
2. These species diffuse outward through a boundary layer along each fiber.
3. These species diffuse outward through the felt.
4. These species diffuse outward through the silicon carbide.

Although the flow rates are slow during the purge, the flow pattern is complex and flow is highly disturbed due to the presence of the felt and the SiC tiles in the flow path.

However, in very general terms, we can treat a diffusion limited flux, J as (ref. 10):

$$J = h(P_s - P_\infty) \approx hP_s \quad (4)$$

Here J is the flux (in weight/unit area-time) of a carbon-containing gas species through a plane away from the surface. P_s is the pressure of that carbon-containing species (e.g., CO or HCN) at the carbon surface, P_∞ is the pressure of that carbon-containing species very far from the surface and can be taken as zero, and h is a mass transfer coefficient. We do not know the form of h , but that is not essential to this analysis. The major factor is the dependence of flux on water vapor content of the gas stream.

Let us assume that reaction (3) is the major route for carbon consumption. This reaction indicates that the vapor flux of carbon-containing species (HCN(g) and CO(g)) is proportional to $[P(\text{H}_2\text{O})]^{1/3}$. Thus decreasing $P(\text{H}_2\text{O})$ from 100 to 10 ppm should decrease the amount of carbon lost by a factor of about 0.32. Decreasing $P(\text{H}_2\text{O})$ from 100 to 1 ppm should decrease the amount of carbon lost by a factor of about 0.1.

DEPOSITION OF CARBON

Another significant observation is the deposition of carbon in the interior holes of some of the graphite blocks. A drop in temperature can lead to carbon deposition. The gaseous products which formed from the nitrogen/water vapor/carbon reactions illustrated in figure 4 were put these back into the code as reactant. In general condensed phase carbon formed at lower temperatures. The graphite walls of the holes provide nucleation sites.

It is likely the carbon which forms in the interior holes is simply the result of decomposition of gases which form at higher temperatures elsewhere in the furnace. This would also explain the substantial deposition of carbon at and near the 'trombone' exit. Any observed deposition in the interior of the hotter blocks may form on furnace heat up, before the interior temperature reaches the exterior temperature. Also, the expected lower partial pressure of H_2O in the interior will promote deposition.

SUMMARY AND CONCLUSIONS

Several conclusions can be drawn from this analysis:

1. The degradation of the graphite blocks and graphite felt is likely due to reaction of carbon with the nitrogen purge gas and water vapor impurities.
2. The reaction products are likely $\text{CN}(\text{g})$, $(\text{CN})_2(\text{g})$, $\text{HCN}(\text{g})$ and $\text{CO}(\text{g})$.
3. The measured 100 ppm water does not appear to be from adsorbed water in the system, but rather a leak from the outside and/or the water cooled induction coils.
4. For the reactions of carbon with nitrogen and water vapor, the flux of carbon is proportional to $[\text{P}(\text{H}_2\text{O})]^{1/3}$. However reactions will still occur with only nitrogen.
5. The deposition of carbon on the inner holes of some blocks is likely due to formation of carbon-containing gases at higher temperatures elsewhere in the furnace and decomposition of these gases in cooler positions on heat-up.

RECOMMENDATIONS

The reactive components of the purge gas should be eliminated or minimized as much as possible. The planned use of an inert purge gas, such as argon or helium, in place of nitrogen should certainly help. The inert gas should be gettered to remove residual oxygen and water vapor. Hot titanium chips are generally used for this purpose. If possible, it would be best to recirculate this gas. This way the product species in the gas stream would suppress further reaction and this would avoid leaking noxious gases into the atmosphere. However, total pressure must be carefully monitored in a recirculating scheme to avoid pressure rises if additional gaseous species are formed.

Nitrogen is, of course, necessary during a test. However the duration of the transition from argon to nitrogen, the test period, and the transition from nitrogen back to argon is a relatively short time compared to the time in a purge condition.

The 100 ppm of H_2O , which appears to arise from a leak in the housing or water cooled power lines, must also be reduced. The calculations indicate that even a reduction to 10 ppm would help substantially. Ideally the level should be below 10 ppm. This may be extremely difficult in a furnace this size. Thorough leak checking of all components should be a part of the reassembly procedure. Monitoring of water vapor throughout the system during purging and testing is essential. A measured level above 10 ppm is an indication of a likely problem.

Although adsorbed water cannot account for all the graphite consumption observed, it can still cause problems. The graphite blocks and felt should be thoroughly outgassed before initial heating. The following heat-up procedure is recommended (ref. 3):

1. Evacuate furnace and begin heating—above boiling point for water, but below the point at which graphite begins to oxidize. About 200 °C would be a good temperature.
2. Back-fill with gettered argon.
3. Evacuate/Back-fill about 3 to 5 times. If the vacuum is better 100 μm Hg or better, then three should be adequate; if the vacuum is closer to 28 mm Hg, then five times is necessary.
4. Flow argon as purge gas in standby condition. If possible, it would be best to recirculate this gas.

As mentioned, the purge gas should be monitored at several points. It is essential to monitor water vapor content, but also gaseous products such as $\text{CN}(\text{g})$, $(\text{CN})_2(\text{g})$, $\text{HCN}(\text{g})$, $\text{H}_2(\text{g})$ and $\text{CO}(\text{g})$ should be monitored. Chilled mirror hygrometers are useful for continuously monitoring water vapor levels. Commercially available quadrupole mass spectrometers, which normally operate in a vacuum, can be adapted to sample the purge gas. Due to the mass spectral overlap of $\text{CO}(\text{g})$ and $\text{N}_2(\text{g})$ (which is always present in some small level, even in Ar purge), an independent $\text{CO}(\text{g})$ analyzer is needed. Another issue is the possible decomposition of the C-N species in a room temperature analyzer, but this is only an issue if a N_2 purge is used.

Ar/H₂O CHEMICAL REACTIONS

If the nitrogen purge gas is replaced with Ar or some other inert gas, the reactions (refs. 1 and 3) which form C-N compounds would be eliminated. However in a furnace this size, there will always be some amount of water vapor. Thus the water-gas equilibrium will always occur (reaction 2). Figure 6(a) to (d) illustrates the effect of an Ar/H₂O purge gas. As expected all of the water vapor reacts completely to CO(g) and H₂(g). H₂(g) is dissociated at the higher temperatures.

Reaction (2) indicates that now the amount of carbon volatilized is proportional to [P(H₂O)]. Since there is no longer a fractional exponent, a decrease in water vapor will have a more significant effect on reducing the amount of carbon volatilized.

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TABLE I.—ESTIMATED AMOUNT OF CARBON CONSUMED FROM THE GRAPHITE BLOCKS. VISUALLY THE BOTTOM TWO BLOCKS SHOWED LITTLE ATTACK. THE 0.06 IN. LOSS MAY BE MANUFACTURING TOLERANCES.

Block	Reduction in diameter, in.	Estimated weight lost, lbs
5	0	0
4E	0.44	296
4D	0.44	296
4C	0.75	507
4B	0.69	464
4A	0.88	590
3D	0.75	507
3C	0.38	254
3B	0.12	85
3A	0	0
2D	0	0
2C	0	0
2B	0	0
2A	0.06	42
1	0.06	42

TABLE II.—CONDITION OF FELT

Location	Condition of felt
Top Block 4E to Bottom Block 3D	Felt slumped, furrowed Inner 2 to 3 in hard with a 'burnt' appearance
Top Block 3C to Bottom Block 2D	Similar to above, but not as severe
Top Block 2C to Bottom Block 1	Felt in good condition

TABLE III.—ESTIMATED TIME AT TEMPERATURE

Year	Time, months	Temperature
1994	~1	3000 °F = 1923 K
1995	~1.5	3000 to 3500 °F = 1923 to 2200 K
1996	~2	3500 °F = 2200 K
1996	~1	4000 to 4500 °F = 2477 to 2755 K

TABLE IV.—FELT CHARACTERISTICS

Fiber diameter	~0.01 mm = 4×10^{-4} in
Estimated density	~0.08 gm/cm ³ = 1 lb/ft ³
Average spacing of fibers	~0.28 mm = 0.011 in
Measured surface area via the BET method (8)	1 m ² /gm = 2.4×10^4 ft ² /lb

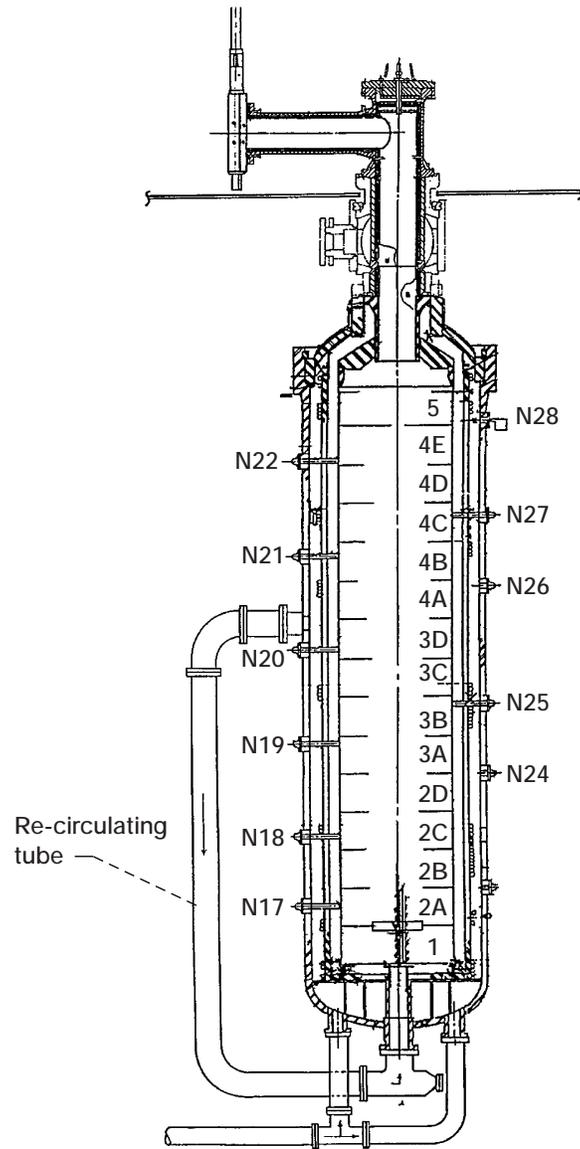


Figure 1.—Schematic of furnace, showing flow path of purge gas.

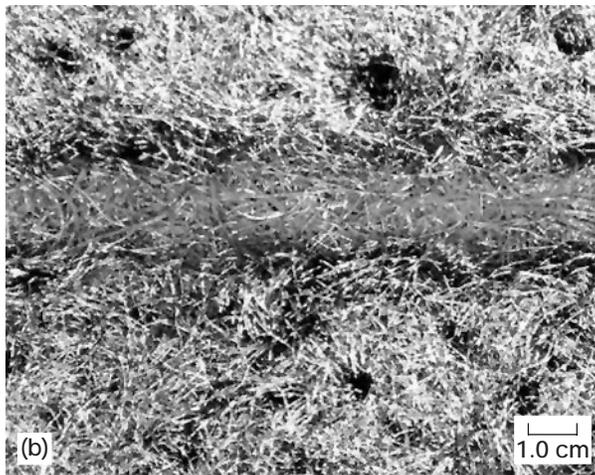


Figure 2.—(a) Photograph of large graphite block. (b) Photograph of the felt, showing grooving.

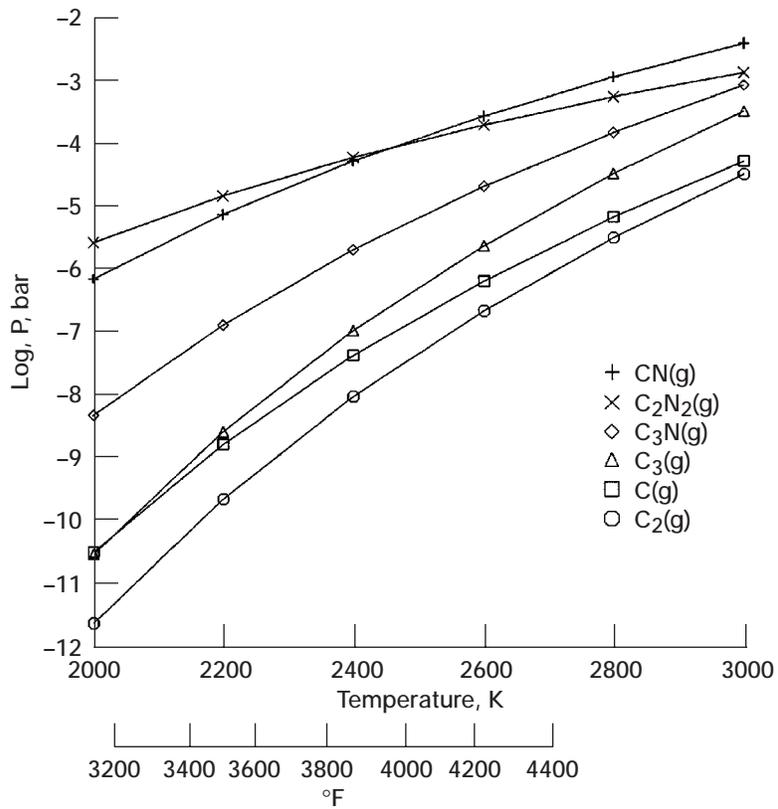


Figure 3.—Calculated reaction products for an infinite amount of carbon, 1.4 bar nitrogen.

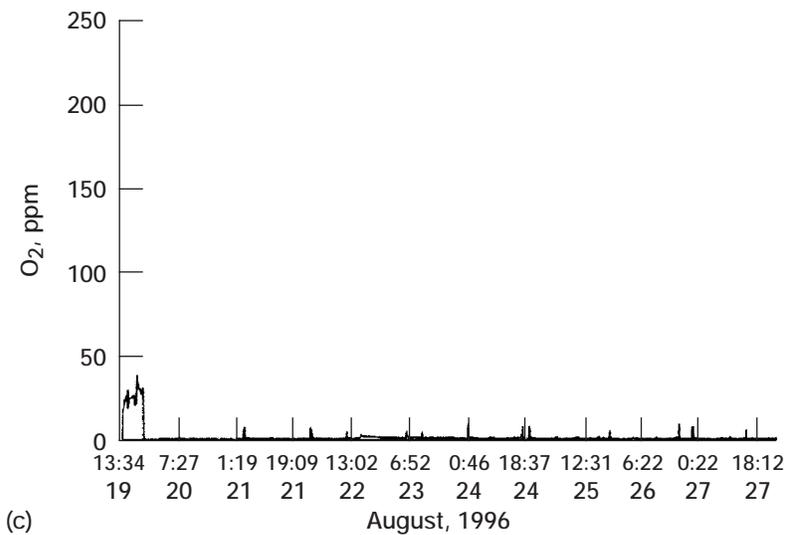
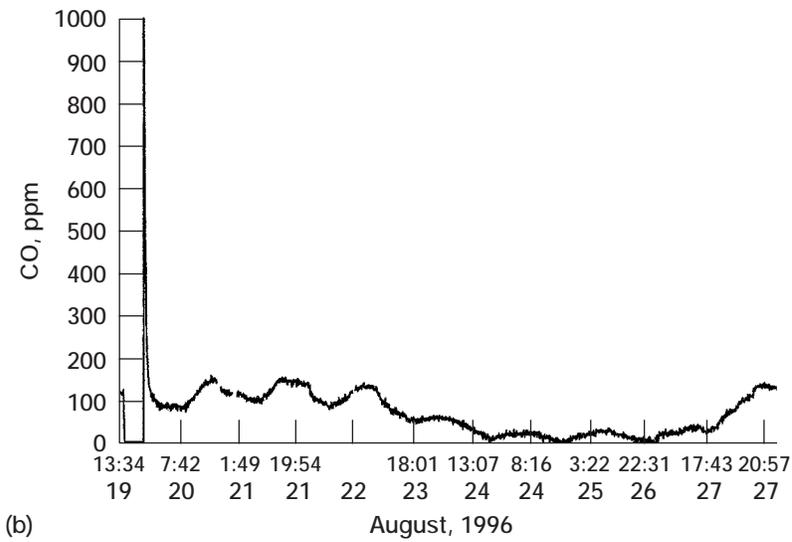
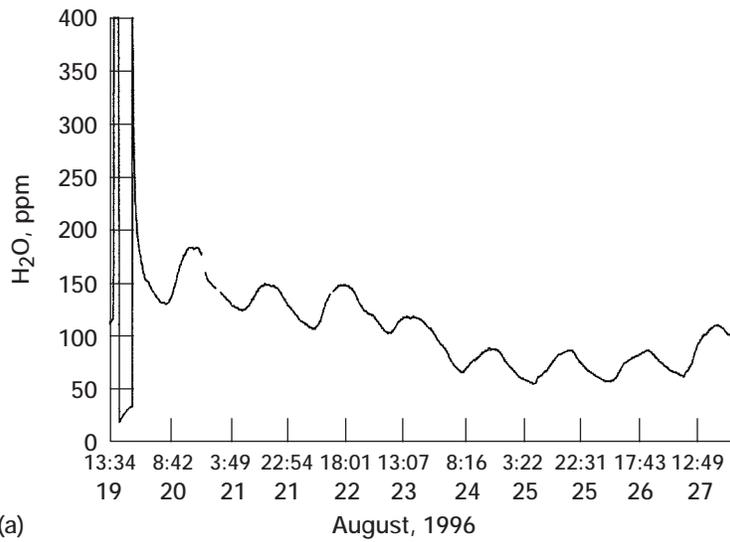


Figure 4.—Gas analyzer (Position N28) output from prior runs, showing that increases in water vapor track with increases in CO(g). (a) H₂O. (b) CO. (c) O₂.

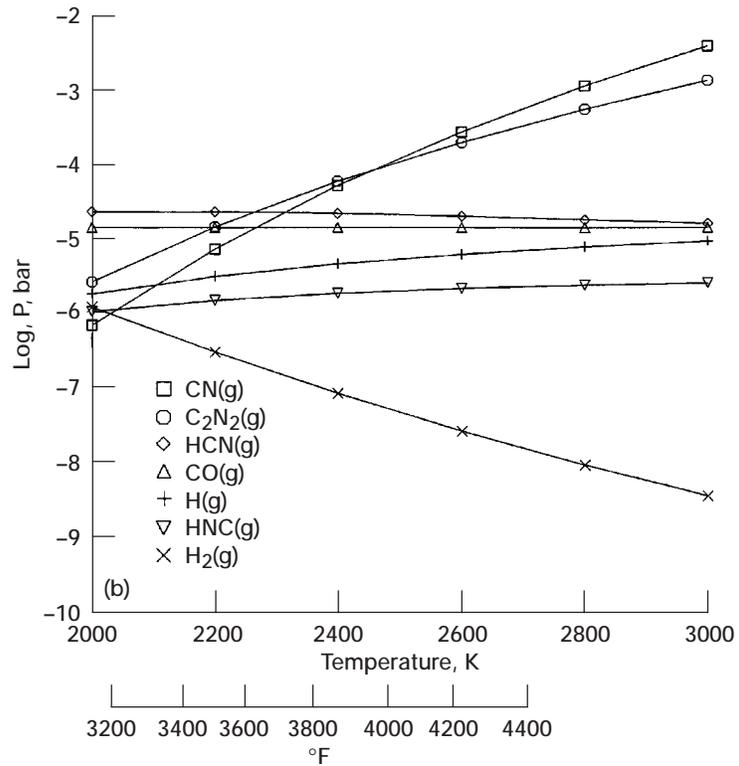
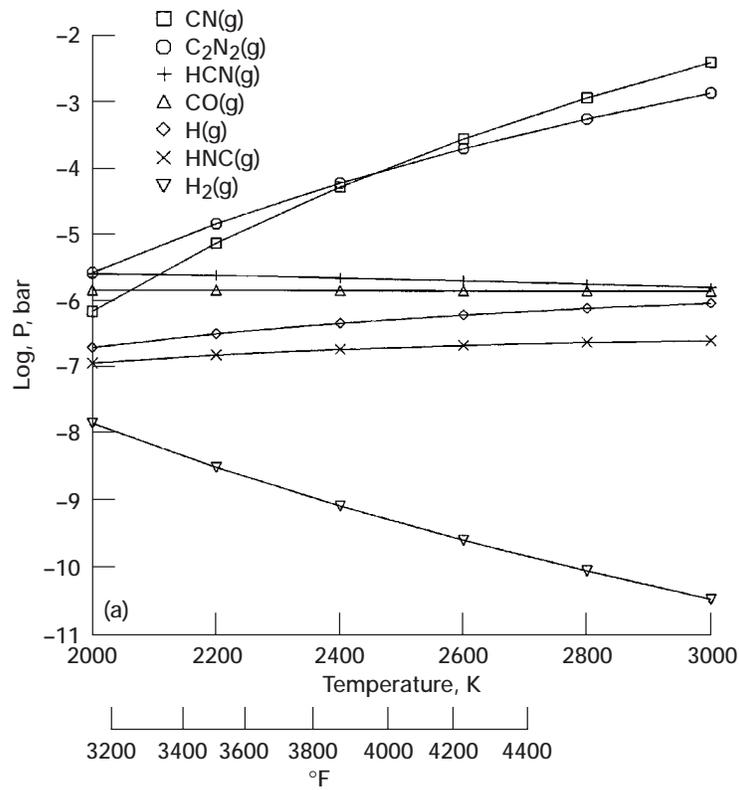


Figure 5.—Calculated reaction products for an infinite amount of carbon, 1.4 bar nitrogen, and varying initial amounts of water vapor. (a) 1 ppm water vapor. (b) 10 ppm water vapor. (c) 100 ppm water vapor. (d) 1000 ppm water vapor.

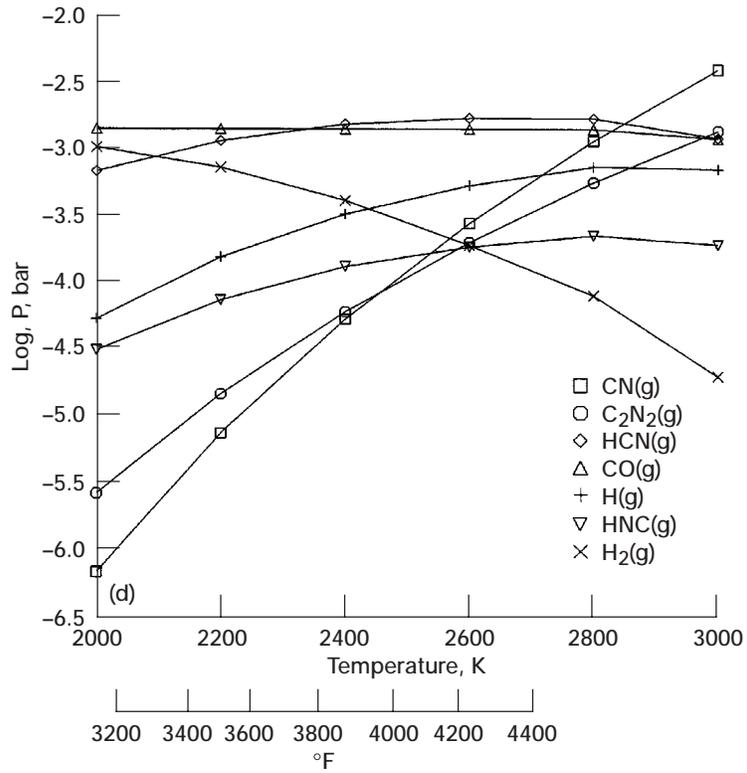
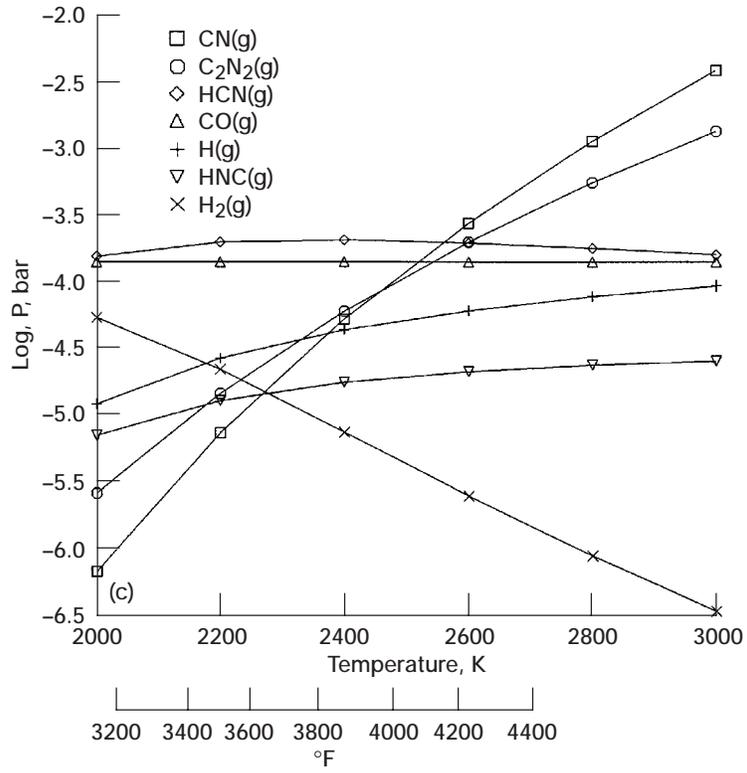


Figure 5.—Concluded. (c) 100 ppm water vapor. (d) 1000 ppm water vapor.

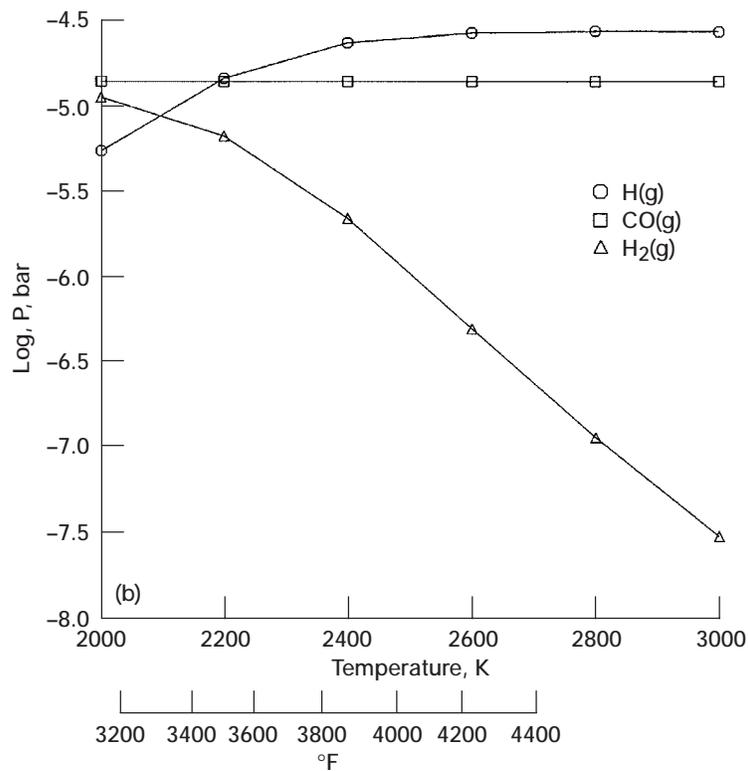
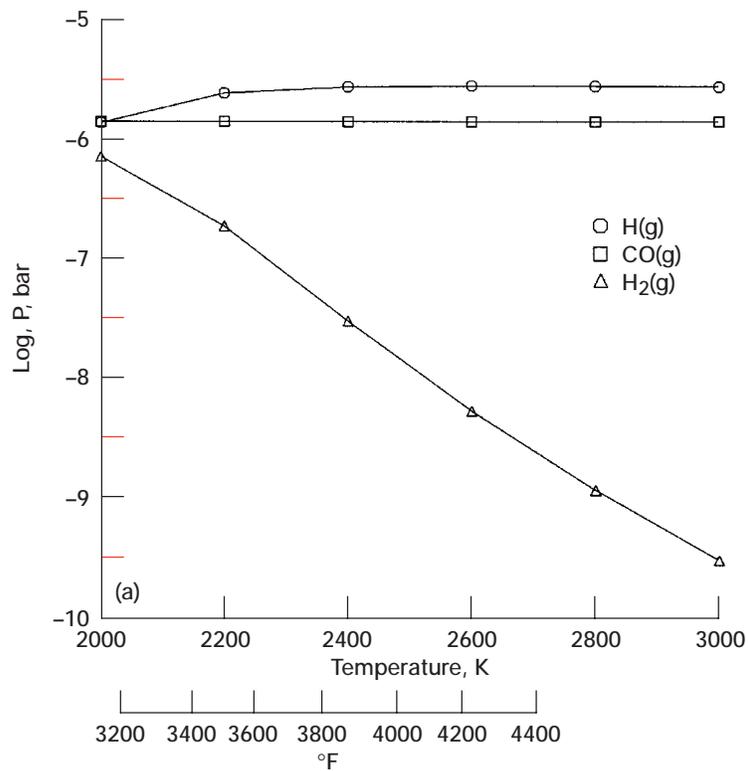


Figure 6.—Calculated reaction products for an infinite amount of carbon, 1.4 bar argon, and varying initial amounts of water vapor. (a) 1 ppm water vapor. (b) 10 ppm water vapor. (c) 100 ppm water vapor. (d) 1000 ppm water vapor.

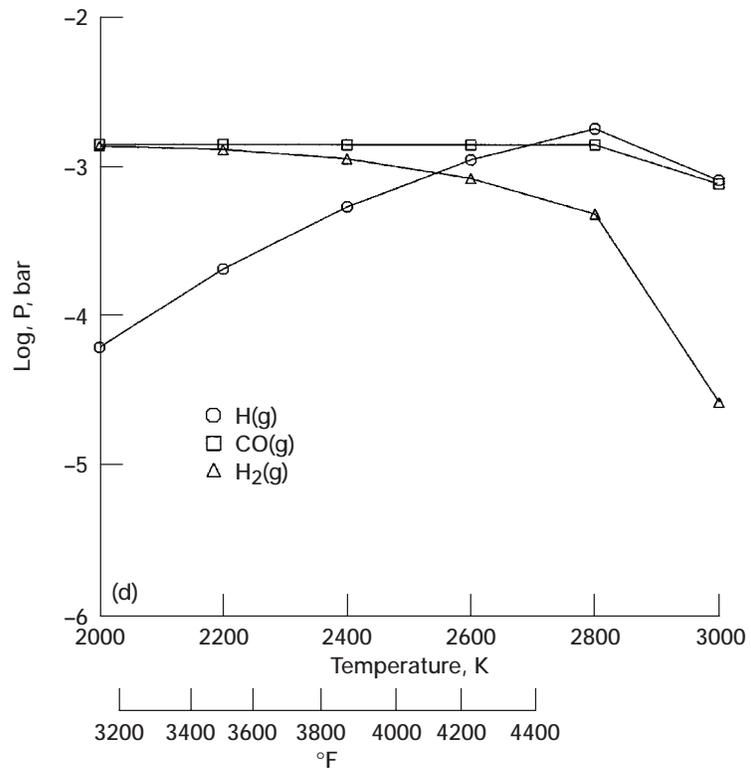
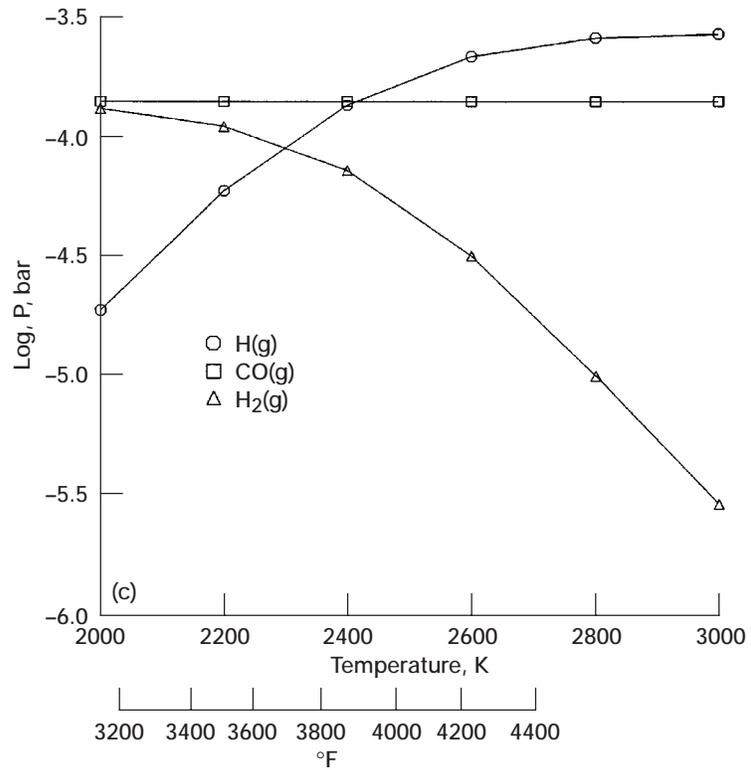


Figure 6.—Concluded. (c) 100 ppm water vapor. (d) 1000 ppm water vapor.

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13. ABSTRACT (Maximum 200 words) A recent rebuild revealed extensive degradation to the large graphite induction furnace in the Hypersonic Tunnel Facility (HTF). This damage to the graphite blocks and insulating felt is examined and modeled with thermochemical equilibrium codes. The primary reactions appear to be with water vapor and the nitrogen purge gas. Based on these conclusions, several changes are recommended. An inert purge gas (e.g. argon or helium) and controlling and monitoring water vapor to about 10 ppm should decrease the damage substantially.				
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